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| Serial No.<br>09/590,496                                | Filing Date June 9, 2000  | E                                   | xaminer                  | Group Art Unit<br>2831 |
| Invention: ASYMMET MANUFACTURE THER                     | RIC ELECTROCHEMICAL SUI   | PERCAPACIT                          | FOR AND METHOD           | OF                     |
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| Patricia 1. 2   | ), Simône   | Dated: D                            | ecember 2, 2002          |                        |

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I certify that this document and fee is being deposited december 2, 2002 with the U.S. Postal Service as first class mail under 37 C.F.R. 1.8 and is addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

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Tracy A. Sweatt

Typed or Printed Name of Person Mailing Correspondence

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| Now You       | FOR:  | ASYMMETRIC ELECTROCHEM<br>SUPERCAPACITOR AND METH<br>MANUFACTURE THEREOF | ,                                  |  |
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APPEAL BRIEF

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#### I. REAL PARTY IN INTEREST

The real party in interest in this appeal is U.S. NANOCORP of Farmington, Connecticut.

#### II. RELATED APPEALS AND INTERFERENCES

There are no other appeals or interferences known to appellant, appellant's legal representatives, or assignee which will directly affect or be directly affected by or have a bearing on the Board's decision in the pending appeal.

#### III. STATUS OF THE CLAIMS

Claims 1-24 are pending in the application. All of the pending claims stand rejected. The rejection of Claims 1-24 is appealed.

#### IV. STATUS OF AMENDMENTS

Per the advisory action mailed September 16, 2002, the amendment submitted on September 3, 2002 was not entered and will not be entered for the purposes of appeal.

An amendment is thus being filed concurrent with this Appeal Brief such that the Specification will be amended to correct certain typographical errors. No Claims are being amended. A copy of the Claims is presented in Appendix A.

#### V. SUMMARY OF THE INVENTION

Supercapacitors (i.e., electrochemical double layer capacitors) store energy by charging of the electrode/electrolyte interface or through faradaic reactions occurring near the electrode surface. One problem with supercapacitors is that while they have high power densities, they often have low energy densities as compared to batteries. It is thus desirable to increase the energy density of supercapacitors without affecting the high power performance. Prior approaches to increase the energy of supercapacitors include the use of non-aqueous electrolytes, the use of conducting polymer electrodes, and the use of mixed oxide pseudo-capacitor electrode materials based on ruthenium oxide and iridium oxide. While these prior approaches offer some advantages, there remains a need for improvements in supercapacitors, particularly asymmetric supercapacitors (i.e., supercapacitors employing materials for both electrodes having an order of magnitude difference in response to an applied voltage).

It has unexpectedly been shown by the Appellants that an asymmetric supercapacitor comprising a positive electrode comprising a current collector and an active material selected from the group consisting of manganese dioxide, silver oxide, iron sulfide and mixtures thereof, a negative electrode comprising a carbonaceous active material and optional current collector, an electrolyte, and a separator plate combines both improved energy density and power density compared to previous designs. The asymmetric supercapacitor of the appealed claims provides improved power density by coupling a positive electrode of high faradaic capacity such as one comprising manganese oxide with a negative electrode that stores charge through charge separation such as one

comprising carbon. Further, when the asymmetric supercapacitor comprises nanostructured electrode materials, improvements in power density are observed.

The dependent claims further define the invention. Manganese dioxide is the preferred active material (Claim 11). The active material can be in a particulate form and can be nanostructured (i.e., having grain sizes of 1 to 100 nanometers) (Claims 12 and 13). The active material can be applied to the electrode by thermal spraying (Claim 14). The carbonaceous material can be a nanofibrous material with particle sizes less than 100 nanometers in diameter (Claims 4-7). In addition to the carbonaceous material, the negative electrode can comprise a current collector such as a metal foil, metal mesh, electrically conductive composite or expanded metal (Claims 2 and 3). The electrolyte can be selected from the group consisting of aqueous electrolyte solutions, organic electrolytes and organic electrolyte solutions (Claim 19). When the electrolyte is an aqueous solution, it can be, selected from for example, aqueous solutions of hydroxides of alkali metals, aqueous solutions of carbonates of alkali metals, sulfuric acid and mixtures thereof (Claim 20).

Example 1 shows the performance including energy densities for supercapacitors according to the appealed claims (p. 14, Examples 1-5). Examples 6 and 7 are comparative examples of symmetric supercapacitors comprising two carbonaceous electrodes (p. 14). While the comparative supercapacitors have energy densities of 0.23 and 1.7 Wh/kg (watt hours/kilogram), the inventive examples having a positive electrode comprising manganese dioxide have energy densities of 2.6 to 7.7 Wh/kg. In addition, Figures 4a and 4b show cyclic voltammograms for inventive examples 1 and 5,

respectively. Both inventive examples show a large window of voltage stability and example 5 shows improved capacitance. Thus, the Examples presented clearly demonstrate that an asymmetric supercapacitor in accordance with the appealed claims shows significant improvements in performance compared to prior art supercapacitors.

In summary, the Appellants have disclosed and claimed an asymmetric supercapacitor that has significant improvements in energy and/or power density as compared to previous supercapacitors. Such asymmetric supercapacitors can be applied, for example, to automotive applications and memory backup devices where both energy density and cost are important factors in the application.

#### VI. ISSUES

- 1. Claims 1, 2, 11-13, 15 and 21 stand rejected under 35 U.S.C. § 102(b) as being allegedly unpatentable over U.S. Patent No. 6,048,645 to Saidi et al. (hereinafter "Saidi").
- 2. Claims 3-4, 8, 10, 14, 16-20 and 22-24 stand rejected under 35 U.S.C. § 103(a) as being allegedly unpatentable over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,953,204 to Suhara et al. (hereinafter "Suhara").

- 3. Claims 5-7 stand rejected under 35 U.S.C. § 103(a) as being allegedly unpatentable over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,953,204 to Suhara et al. and in further view of U.S. Patent No. 6,031,711 to Tennent et al. (hereinafter "Tennent").
- 4. Claim 9 stands rejected under 35 U.S.C. § 103(a) as being allegedly unpatentable over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,415,959 to Pyszczek et al. (hereinafter "Pyszczek").

## VII. GROUPING OF CLAIMS

The claims stand together.

#### VIII. ARGUMENT

1. Claims 1, 2, 11-13, 15 and 21 are patentable under 35 U.S.C. § 102(b) over Saidi.

Instant independent Claim 1 is directed to an asymmetric supercapacitor comprising a positive electrode comprising a current collector and an active material selected from the group consisting of manganese dioxide, silver oxide, iron sulfide and mixtures thereof, a negative electrode comprising a carbonaceous active material and an optional current collector, an electrolyte and a separator plate. The claimed supercapacitor that has improved energy and power densities compared to prior art supercapacitors. Instant independent Claim 21 is similar to Claim 1 except the active material is manganese dioxide. Appellants note that the positive electrode (i.e., the cathode) comprises the active material of instant Claim 1 (i.e., manganese dioxide).

Saidi teaches a lithium-ion battery which comprises in part a positive electrode having an active material comprising the lithium-rich manganese oxide tetragonal structure Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> (Column 2, lines 45-48). In Column 7, line 18 to Column 8, line 11, the synthesis of the lithium-rich manganese oxide is detailed. In the synthesis, a manganese compound (i.e., βMnO<sub>2</sub>) and a lithium compound (LiOH, LiNO<sub>3</sub>, etc.) are clearly disclosed as chemical precursors to the formation of Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> (Column 7, lines 18-22 and Column 8, lines 5-11). It is further disclosed that to form the required βMnO<sub>2</sub>, electrolytic manganese dioxide (EMD) (i.e., gamma EMD) can be deposited on the anode of an electrolysis cell (Column 7, lines 40-48). Appellants note that the gamma EMD of Saidi is deposited on the anode or negative electrode of the electrolysis cell (Column 7, lines 47-48). It is not disclosed in Saidi what the cathode or positive electrode used in this electrolysis reaction comprises.

Saidi further teaches that, once formed, the electrochemical manganese dioxide (EMD) is *removed* from the anode, crushed, ground, washed, neutralized and dried in a rotary dryer (Column 7, lines 50-52). The EMD is then used to form the preferred βMnO<sub>2</sub> *precursor* that is then used to react with the lithium compound by heating the EMD to a temperature of about 400°C resulting in the desired Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub>(Column 7, lines 31-37). The electrode taught in Saidi is then made by coating a mixture of a binder, the active material, i.e., Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub>, and a carbon powder onto a current collector (Column 8, lines 65-67). The positive electrode, therefore, comprises the lithium manganese oxide active material (Column 8, line 67 – Column 9, line 1), and not manganese dioxide. The

manganese dioxide disclosed in Saidi is clearly a chemical precursor to a component of the anode.

In the Office Action dated July 1, 2002, the Examiner alleged in part that Saidi discloses an electrochemical cell comprising a positive electrode comprising a current collector and an active material selected from the group consisting of manganese dioxide. In the Advisory Action dated September 16, 2002, the Examiner maintains the rejection and alleges that the "Applicants have argued on 9/3/2002 that Saidi et al. do not disclose the anode electrode comprising manganese dioxide" (Paper 14, continuation of line 5). Appellants submit that the Examiner has misconstrued the Appellants' arguments. In the Amendment dated September 3, 2002, Appellants clearly argue that the manganese dioxide of Saidi is on the anode (i.e., negative electrode) and not on the cathode (i.e., positive electrode) as required by the instant claims (September 3, 2002 Amendment, Page 6).

To anticipate a claim under 35 U.S.C. § 102, a single source must contain all of the elements of the claim. Lewmar Marine Inc. v. Barient, Inc., 827 F.2d 744, 747, 3 U.S.P.Q.2d 1766, 1768 (Fed. Cir. 1987), cert. denied, 484 U.S. 1007 (1988).

Appellants submit that Saidi does not disclose manganese dioxide on the positive electrode (i.e., cathode) as required by the instant claims. First, in the July 1, 2002 Office Action, the Examiner alleges that Saidi discloses a positive electrode comprising manganese dioxide as an active material and cites Column 2, lines 46-48 (Paper 14, Page 3). Appellants submit that the Examiner is mistaken in his reading of Saidi. In Column

2, lines 46-48, Saidi discloses a positive electrode comprising Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub>. The distinction between the Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> of Saidi and the MnO<sub>2</sub> of the instant claims is highlighted in Saidi at Column 7, lines 18-22 and Column 8, lines 5-11 where manganese dioxide is clearly disclosed as a chemical precursor to Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub>. Thus, Saidi at Column 2, lines 46-48 does not disclose a positive electrode comprising MnO<sub>2</sub> as alleged by the Examiner.

Second, as pointed out by the Examiner in the September 13, 2002 Advisory

Action, the manganese dioxide disclosed in Saidi is deposited on the anode. For

clarification, Saidi clearly defines the anode as the negative electrode and the cathode as
the positive electrode (Column 1, lines 19-24). Thus, the manganese dioxide coating of
Saidi is on the anode or negative electrode and not on the positive electrode as required
by the instant claims. As discussed above, this manganese dioxide is not a part of the
final electrochemical cell, but is a chemical precursor to form the Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> that is a part
of the electrochemical cell of Saidi. Thus, there is at least one element of the instant
claims (a positive electrode comprising manganese dioxide) that is not disclosed in Saidi.

Thus Saidi cannot anticipate the instant claims.

For at least these reasons, Appellants request reversal of the Examiner's rejections of Claims 1, 2, 11-13, 15 and 21 and allowance of Claims 1, 2, 11-13, 15 and 21.

2. Claims 3-4, 8, 10, 14, 16-20 and 22-24 are patentable under 35 U.S.C. § 103(a) over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,953,204 to Suhara et al. (hereinafter "Suhara").

Claims 3-4, 8, 10 14 and 16-20 are dependent upon independent Claim 1 and Claims 22-24 are dependent upon independent Claim 21 and further limit the independent

Claims. As discussed above, Saidi cannot anticipate instant independent Claims 1 and 21 because Saidi is missing the element of a positive electrode comprising manganese dioxide. Appellants submit that Suhara does not cure the defects of Saidi.

Suhara teaches and claims an electric double layer capacitor having a positive electrode comprising activated carbon and a negative electrode having a current collector of porous metal incapable of forming an alloy with lithium (Abstract). Relevant to the instant dependent claims, the Examiner cites several passages in Suhara as allegedly disclosing various claim elements. In at least one case, Appellants disagree with the Examiner's analysis. While Suhara discloses many forms of carbon materials in Column 6, lines 4-10, there is no disclosure of a nanofibrous carbonaceous material. There is no disclosed diameter of the carbon fibers and thus no disclosure of nanofibrous carbon fibers. This point is moot, however, because Suhara provides no teaching regarding a positive electrode comprising manganese dioxide. Thus, Appellants submit that Suhara cannot cure the defects of Saidi.

For an obviousness rejection to be proper, the Examiner must meet the burden of establishing a prima facie case of obviousness. *In re Fine*, 5 U.S.P.Q.2d 1596, 1598 (Fed. Cir. 1988). Establishing a prima facie case of obviousness requires that all elements of the invention be disclosed in the prior art. *In Re Wilson*, 165 U.S.P.Q. 494, 496 (C.C.P.A. 1970).

As described in detail above, Saidi fails to disclose a positive electrode comprising manganese dioxide and thus fails to anticipate the instant Claims. Suhara does not supply the missing claim element. There is no teaching in Suhara regarding

electrodes comprising manganese dioxide. Thus, Saidi and Suhara, alone and in combination, fail to render the instant Claims obvious. Further, Claims 3-4, 8, 10, 14, 16-20 and 22-24 depend from allowable independent Claims 1 and 21 and are themselves allowable.

For at least these reasons, Appellants request reversal of the Examiner's rejections of Claims 3-4, 8, 10, 14, 16-20 and 22-24 and allowance of Claims 3-4, 8, 10, 14, 16-20 and 22-24.

3. Claims 5-7 are patentable under 35 U.S.C. § 103(a) over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,953,204 to Suhara et al. and in further view of U.S. Patent No. 6,031,711 to Tennent et al.

Claims 5-7 are dependent upon independent Claim 1 and further limit independent Claim 1. As discussed above, Saidi cannot anticipate instant independent Claim 1 because Saidi is missing the instant Claim element of a positive electrode comprising manganese dioxide. Appellants submit that Suhara and Tennent do not cure the defects of Saidi.

As discussed above, Suhara does not provide the missing claim element of a positive electrode comprising manganese dioxide and cannot render the instant claims obvious, alone or in combination with Saidi. Tennent does not cure the defects.

Tennent teaches and claims the use of graphitic nanofibers in electrochemical capacitors (Abstract). Tennant does disclose fibers of diameter 3.5 to 70 nanometers (Column 9, lines 49-54). Tennent, however, provides no teaching regarding an electrode

comprising manganese dioxide. Thus, Appellants submit that Tennent cannot cure the defects of Saidi and Suhara.

Saidi, Suhara and Tennent, alone or in combination, fail to disclose a positive electrode comprising manganese dioxide. Because the references fail to teach at least one instant claim element, the references fail to render the instant claims obvious. Further, Claims 5-7 depend from allowable independent Claim 1 and are thus themselves allowable.

For at least these reasons, Appellants request reversal of the Examiner's rejections of Claims 5-7 and allowance of Claims 5-7.

4. Claim 9 is patentable under 35 U.S.C. § 103(a) over U.S. Patent No. 6,048,645 to Saidi et al. in view of U.S. Patent No. 5,415,959 to Pyszczek et al. (hereinafter "Pyszczek").

As discussed above, Saidi cannot anticipate instant independent Claim 1 because Saidi is missing the instant Claim element of a positive electrode comprising manganese dioxide. Applicants submit that Pyszczek does not cure the defects of Saidi.

As discussed above, Saidi discloses Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> as the active material for the positive electrode. The negative electrode may be metallic lithium but is preferably an intercalation active material such as metal oxide or graphite (Column 9, lines 27-41). Saidi does not disclose the use of manganese dioxide, silver oxide, iron sulfide and mixtures thereof as active material in the positive electrode as claimed in the instant Application.

Pyszczek teaches and claims an electrochemical cell comprising a separator comprising a fabric woven from a halogenated polymeric fiber (Abstract). As far as appellants are aware, Pyszczek does not disclose a carbonaceous woven mat as alleged by the Examiner. The passages referred to by the Examiner in Paper 4 Pages 6-7 refer to the separator, not the negative electrode. The electrochemical cell comprises an anode (negative electrode) made of metals from Groups IA, IIA, and IIIB of the Periodic Table such as lithium typically as a thin sheet or foil and a current collector (Column 4, lines 10-33). The cathode (positive electrode) may be made of manganese dioxide, iron sulfide or other metal compounds (Column 4, lines 23-27). The cathode can be pressed into a pellet with a material having electronic conduction characteristics such as carbon (Column 4, lines 36-38).

An Examiner cannot establish obviousness by locating references that describe various aspects of a patent applicant's invention without also providing evidence of the motivating force, which would have impelled one skilled in the art to do what the patent applicant has done. *Ex parte Levengood*, 28 U.S.P.Q. 1300 (Bd. Pat. App. Int. 1993). The references, when viewed by themselves and not in retrospect, must suggest the invention. *In Re Skoll*, 187 U.S.P.Q. 481 (C.C.P.A. 1975).

Saidi discloses only one useful active material for the positive electrode,  $Li_2Mn_2O_4$ . Saidi does not teach or suggest that any other material would be useful as the positive electrode active material. Pyszczek does not teach or suggest that manganese dioxide or iron sulfide can be used in place of  $Li_2Mn_2O_4$ . In addition, Pyszczek does not teach a positive electrode comprising manganese dioxide or iron sulfide in combination

with a negative electrode comprising a carbonaceous active material. The negative electrode of Pyszczek is a metal selected from Groups IA, IIA, and IIIB of the Periodic Table. Thus, Pyszczek does not provide the motivation to use a positive electrode comprising manganese dioxide or iron sulfide with a carbonaceous negative electrode, nor does it provide motivation to replace the Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> of Saidi with manganese dioxide or iron sulfide. Therefore Appellants respectfully assert that there is no motivation in the references themselves to combine the positive electrode active material of Pyszczek with Saidi; therefore, the combination of references cannot render the instant Application obvious. Further, Claim 9 depends from allowable independent Claim 1 and is itself allowable.

For at least these reasons, Appellants request reversal of the Examiner's rejections of Claim 9 and allowance of Claim 9.

#### IX. CONCLUSION:

In view of the foregoing, it is urged that the final rejection of Claims 1-24 be overturned and the Claims allowed. The final rejection is in error and should be reversed.

If there are any additional charges with respect to this Brief, please charge them to

Deposit Account No. 06-1130 maintained by Applicants' attorneys.

Respectfully submitted,

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### Appendix A- Clean Claims

Claim 1. An asymmetric supercapacitor comprising;

a positive electrode comprising a current collector and an active material selected from the group consisting of manganese dioxide, silver oxide, iron sulfide and mixtures thereof;

a negative electrode comprising carbonaceous active material;

an electrolyte; and

a separator plate.

- Claim 2. The asymmetric supercapacitor of Claim 1 wherein the negative electrode further comprises a current collector.
- Claim 3. The asymmetric supercapacitor of Claim 2 wherein the current collector is selected from the group consisting of metal foil, metal mesh, electrically conductive polymer composites and expanded metal.
- Claim 4. The asymmetric supercapacitor of Claim 1 wherein the carbonaceous active material comprises nanofibrous materials.
- Claim 5. The asymmetric supercapacitor of Claim 4 wherein the carbonaceous active material is discrete carbon fibers less than 10 microns in diameter.
- Claim 6. The asymmetric supercapacitor of Claim 5 wherein the carbonaceous active material is carbon fibers less than 100 nm in diameter.
- Claim 7. The asymmetric supercapacitor of Claim 6 wherein the carbonaceous active material is carbon fibers less than 50 nm in diameter.
- Claim 8. The asymmetric supercapacitor of Claim 1 wherein the negative electrode has a thickness about 50 microns to about 375 microns.

Claim 9. The asymmetric supercapacitor of Claim 1 wherein the carbonaceous active material is non-woven mat, woven cloth or two dimensional sheet comprising carbonized polymer.

- Claim 10. The asymmetric supercapacitor of Claim 1 wherein the negative electrode further comprises a collection coating.
- Claim 11. The asymmetric supercapacitor of Claim 1 wherein the positive electrode active material comprises manganese dioxide.
- Claim 12. The asymmetric supercapacitor of Claim 11 wherein the manganese dioxide is nanostructured.
- Claim 13. The asymmetric supercapacitor of Claim 1 wherein the positive electrode active material is nanostructured.
- Claim 14. The asymmetric supercapacitor of Claim 1 wherein the active material is applied to the current collector by thermal spray.
- Claim 15. The asymmetric supercapacitor of Claim 1 wherein the positive electrode further comprises a binder.
- Claim 16. The asymmetric supercapacitor of Claim 1 wherein the current collector is selected from the group consisting of metal foil, metal mesh, electrically conductive polymer composites and expanded metal.
- Claim 17. The asymmetric supercapacitor of Claim 1 wherein the positive electrode thickness is less than about 250 microns.
- Claim 18. The asymmetric supercapacitor of Claim 17 wherein the positive electrode thickness is less than about 50 microns.

Claim 19. The asymmetric supercapacitor of Claim 1 wherein the electrolyte is selected from the group consisting of aqueous electrolyte solution, organic electrolyte and organic electrolyte solution.

Claim 20. The asymmetric supercapacitor of Claim 19 wherein the aqueous electrolyte is selected from the group consisting of aqueous solutions of hydroxides of alkali metals, aqueous solutions of carbonates of alkali metals, sulfuric acid and mixtures thereof.

## Claim 21. An asymmetric supercapacitor comprising;

a positive electrode comprising a current collector and manganese dioxide;
a negative electrode comprising carbonaceous active material;
an aqueous electrolyte solution; and a separator plate.

- Claim 22. The asymmetric supercapacitor of Claim 21 wherein the carbonaceous active material is nanofibrous.
- Claim 23. The asymmetric supercapacitor of Claim 21 wherein the manganese dioxide is nanostructured.
- Claim 24. The asymmetric supercapacitor of Claim 21 wherein the carbonaceous active material is nanofibrous and the manganese dioxide is nanostructured.